Japanese Published Unexamined (Kokai Koho) Patent Application (A) No. 04-028786, published January 31, 1992; Application No. 02-133281, filed May 22, 1990; Int.Cl.: C 09K 11/08, 11/00, 11/56; Inventor(s): Ryoichi Takeda et al.; Assignee: Kansai Nippon Electric Corporation; Japanese Title: Method to Manufacture Diffusion Type EL

METHOD TO MANUFACTURE DIFFUSION TYPE EL

CLAIM(S)

A method to manufacture a fluorescent body for a diffusion type EL characterized by its comprising the following steps: a step of adding an activating agent and a co-activating agent to ZnS; a step of sintering the admixture of ZnS by using a reactor having a multilayer structure in a furnace at a high temperature; a step of supplying sulfide, such as ZnS or sulfur powder, into the outer tank unit of the reactor when the sintering is performed in the atmospheric air at a high temperature.

DETAILED DESCRIPTION OF THE INVENTION

(Field of Industrial Application)

The present invention pertains to a method to manufacture a fluorescent body for a high brightness diffusion type EL.

(Prior Art)

The prior art fluorescent body for a diffusion type EL was manufactured by mixing an activating agent, Cu, and a co-activating agent, Al, Cl, or Br, into ZnS,

and by sintering at a high temperature in the atmosphere selected out of H_2S, Ar, N_2, and air in a furnace by using a reactor shown in Fig. 5.

(Problems of the Prior Art to Be Addressed)

H_2S, however, is a harmful gas and requires a treatment facility. Although N_2 and Ar are inactive gases, they cause high manufacturing cost of a fluorescent body, as they are supplied to the furnace at a constant flow rate, which is a problem.

When the fluorescent body is sintered in the furnace, a luminescent center is formed in the fluorescent body, and ZnO is formed on the surface of the fluorescent body, reducing the luminescent efficiency, so a process of etching it away with HCL was required (Japanese Examined Patent Application 59-16399).

(Means to Solve the Problem)

The present invention, to solve the problem of high manufacturing cost of the prior art fluorescent body, presents a manufacturing method of fluorescent body for a diffusion type EL, whereby the reactor for sintering ZnS in the high temperature air is made into a multilayer structure, and sulfide, such as sulfur powder or ZnS, is supplied into its outer tank unit in order to eliminate said etching process.

(Operation)

When a fluorescent body is sintered at a high temperature, a sulfide, such as sulfur powder or ZnS powder, is supplied to the outer tank unit of the reactor. By so doing, the sulfur atmosphere is created inside the reactor during the sintering, preventing ZnO from being formed on the surface of the fluorescent body.

Therefore, the harmful gas needs not be used, and a highly bright fluorescent body can be manufactured without the process of etching.

(Embodiment Example)

One example of the embodiment of the present invention is explained below by an example of using ZnS, Cu, and Br in the method of manufacturing a fluorescent body, with reference to Fig. 1. Into 100 g of ZnS as the raw material powder, 1.54 g (0.75 mol%/ZnS) of Cu as the activating agent was added in form of $Cu(cH_3coo)_2H_2O$, and 9.65 g (9.6 mol%/ZnS) of Br as the co-activating agent was added in form of NH_4Br. Ethanol was further added to this admixture to make it into a slurry state. The admixture was dried in an oven at 100 C for 2 hours. This admixture defined here as raw material 1 was put into the inner tank 4 of highly pure alumina crucible 3 having a double-layer structure; 20 g of sulfur powder was supplied to the outer tank unit 5 and the cover was put on; the raw material 1 was sintered at 1000 C in the atmospheric air for 8 hours in the sintering furnace. After the sintering has been completed, the sinter was cooled in the furnace and was removed out of the furnace when its temperature was dropped to a normal temperature.

An excessive amount of sulfide, Cu, was attached to the fluorescent body surface after the sintering, so this sinter was immersed into a 10 wt% aqueous solution alkalified with 5 wt% NaoH and agitated for cleaning at 50 C for 1 hour.

Then, the sinter was cleaned with pure water until pH became 7; the water was substituted with ethanol; the sinter was put to ultrasonic treatment for loosen the agglutinated particles.

The fluorescent body was thus made by using ZnS, Cu, and Br, was driven by 400 Hz of alternating current. Its brightness level vs. voltage characteristics were examined and compared with those of the prior art fluorescent body, which is shown in Fig. 2 in form of a graph.

The prior art fluorescent body which has not been put to the etching treatment had a very low brightness level, and the prior art fluorescent body which has been put to the etching treatment to remove the Zno on the surface had a higher brightness level. The fluorescent body of the present invention had a high brightness level without the etching treatment since it was sintered in the sulfur atmosphere and the ZnO formation on the surface was prevented. Moreover, the fluorescent body of the present invention had the brightness level 10% higher than the prior art had. The probable reason for this is that, with the prior art fluorescent body, its luminous efficiency was reduced at the time of etching since the ZnO on the surface is not only etched away at the time of etching but the ZnS matrix is also etched to a certain extent.

Fig. 3 (a), (b) show the X-ray refraction patterns of the fluorescent body of the present invention and that of the prior art (not etched on the surface), respectively.

It is evident from these patterns that the ZnO is not formed on the fluorescent body of the present invention.

The embodiment of the present invention is not limited to the example of the structure shown in Fig. 1. It is also possible that the sulfur powder is provided on the surface of the raw material to form the sulfur atmosphere.

In the above example, the fluorescent body using ZnS, Cu, and Br was explained, but the same result will be produced even if the co-activating agent is changed to Al, Xl, or I.

(Advantage)

As explained above, in the present invention, by making the reactor in multilayer structure and by providing sulfide to form the sulfur atmosphere during the sintering, the etching treatment in the gas during the sintering can be eliminated. In addition, a fluorescent body having a higher brightness level than the prior art fluorescent body can be manufactured, which is an advantage.

Moreover, in the prior art fluorescent body manufacturing method, the fluorescent body sintered near the upper part of the reactor and near the wall of the reactor had a weak photo-luminescent section, so the fluorescent body sintered in the center section of the reactor was selectively used. In the method of the present invention, uniform sintering can be performed, so every portion of the sinter can be used, improving the yield in production, which is advantageous.

BRIEF DESCRIPTION OF THE DRAWINGS

Fig. 1 illustrates a sectional view of the reactor used for explaining the embodiment of the present invention.

Fig. 2 shows the relationship between the brightness level and the voltage in the EL elements manufactured by using the fluorescent body of the present invention and of the prior art.

Fig. 3 (a), (b) show the X-ray refraction patterns of the flourescent body of the present invention and that of the prior art (with no etching treatment).

Fig. 4 shows another embodiment example of the present invention. Fig. 5 shows the example of the prior art fluorescent body.